

# Characterization of the Pesticide Chlordane in Estuarine River Sediments

Y. Ouyang,\* L.-T. Ou, and G. C. Sigua

## ABSTRACT

Sediments are increasingly recognized as both carrier and potential source of contaminants in aquatic environments. This study investigated the characteristics and spatial distribution of total chlordane and its three most abundant compounds, including  $\alpha$ -chlordane,  $\gamma$ -chlordane, and *trans*-nonachlor, in sediments from the Cedar and Ortega rivers, Florida, USA, using geographic information system (GIS)-based kriging analyses and field measurements. Kriging analysis showed that two areas, one from the Cedar River area and the other from the northern end of the Ortega River area, were contaminated. The maximum concentrations of total chlordane,  $\gamma$ -chlordane,  $\alpha$ -chlordane, and *trans*-nonachlor in the sediments were, respectively, 101.8, 20.1, 26.3, and 19.2  $\mu\text{g/kg}$ . A plot of total organic carbon (TOC)-normalized chlordane concentrations showed that effects of grain size on sediment chlordane contamination were negligible. A principal axis analysis further revealed that a linear correlation existed between  $\alpha$ -chlordane and total chlordane as well as between  $\gamma$ -chlordane and total chlordane, whereas no correlation existed between *trans*-nonachlor and total chlordane. Comparison of total chlordane concentration with Florida Sediment Assessment Guidelines showed that the Cedar River and the northern end of the Ortega River had total chlordane concentrations above the probable effect level (4.79  $\mu\text{g/kg}$ ), which could pose a potential risk to aquatic life.

ORGANIC CONTAMINANTS are ubiquitous in surface environments throughout the world. Most of these contaminants are highly hydrophobic and persist in sediments of rivers and lakes long after their release. A well-known organic contaminant is the pesticide chlordane. Chlordane was widely used in the United States for both agricultural and residential applications from the time of its introduction in the 1940s through its suspension by the USEPA in 1988 (Ahrens et al., 1962; USEPA, 1990). As applied, chlordane was not a single compound but a mixture of more than 140 different related compounds known as technical chlordane. The three most abundant compounds of technical chlordane, *cis*- (or  $\alpha$ ) chlordane, *trans*- (or  $\gamma$ ) chlordane, and *trans*-nonachlor, could account for approximately 25% (Eitzer et al., 2001), 51% (Falandysz et al., 2001), or 95% (Mattina et al., 1999) of the technical chlordane, depending on manufacturer. Although a ban on chlordane application was imposed in the United States in 1988, this chlorinated hydrocarbon still exists in the environment and in biota (Falandysz et al., 2001; Bidleman et al., 2002). The lipophilic character of chlordane facilitates its accumulation

and persistence in lipid-rich tissues of biota, and its biomagnification on the food chain is a major concern.

Several studies have been devoted to investigating the fate of chlordane in the soil, water, and air system. Eitzer et al. (2001) employed chiral gas chromatography in conjunction with ion trap mass spectrometry to measure the compositional chiral profiles of weathered soil residual chlordane. These authors found that biotic degradation and abiotic weathering of chlordane occurred in the soils used in their studies. Gundersen et al. (2000) reported that 5 of 10 Ohio River paddlefish egg samples exceeded the Food and Drug Administration's action limit for chlordane (0.3  $\mu\text{g/g}$ ).

Human activities such as agricultural, residential, and commercial pesticide applications are responsible for elevated technical chlordane levels in sediments from the Cedar and Ortega rivers located in the St. Johns River watershed, Florida. Technical chlordane entering the Cedar and Ortega rivers from upstream can remain in the water column adsorbed to suspended solids and can transport downstream with normal water flow. Restoration and protection of the Cedar and Ortega rivers have been a challenge. During its 1987 session, the Florida Legislature enacted the Surface Water Improvement and Management (SWIM) Act, which identified this segment of the St. Johns River as one of the areas of critical concern. To restore the environmental health of the rivers, the St. Johns River Water Management District (SJRWMD) is currently evaluating the feasibility of removing sediments and associated contaminants (e.g., heavy metals and hydrocarbons) by dredging. To ensure cost-effective remediation, a quantitative study of the contaminants including chlordane in the river sediments is crucial.

The purpose of this study was to investigate the characteristics and spatial distribution of pesticide chlordane in sediments from the Cedar and Ortega rivers, using GIS-based kriging analyses and field measurements. The specific objectives were to (i) examine the spatial distribution of  $\alpha$ -chlordane,  $\gamma$ -chlordane, and *trans*-nonachlor in the sediments from the Cedar and Ortega rivers using GIS-based kriging analysis and field measurements; (ii) characterize the relationships among the chlordane species; and (iii) determine the potential risk of the sediment chlordane to aquatic life using Florida Sediment Assessment Guidelines.

## MATERIALS AND METHODS

### Study Site

The Cedar and Ortega rivers basin is located in south-central Duval County and is a tributary of the St. Johns River,

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**Abbreviations:** GIS, geographic information system; PAA, principal axis analysis; PEL, probable effect level; TEL, threshold effect level; TOC, total organic carbon.

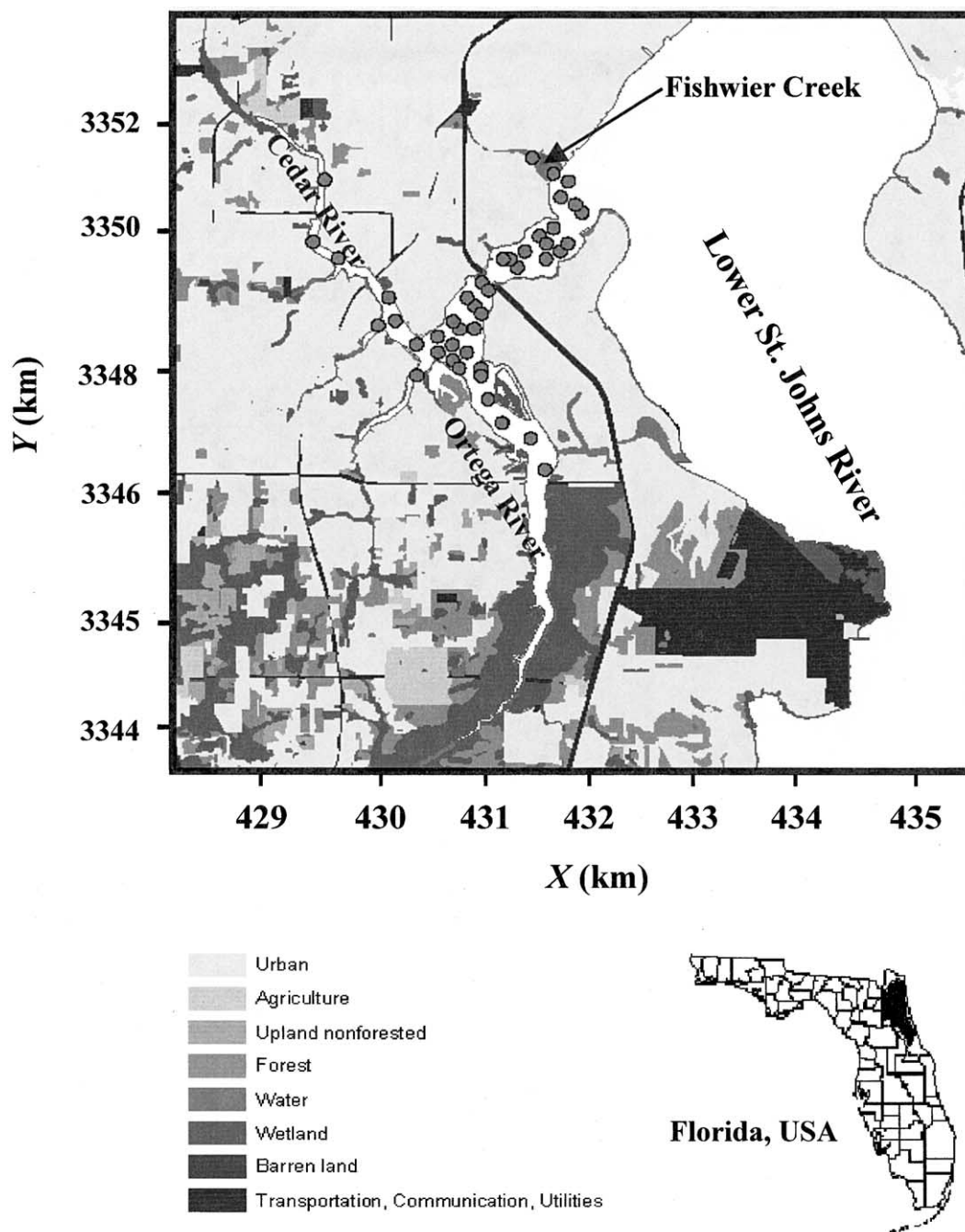


Fig. 1. Location of the study area showing the Cedar and Ortega rivers watershed and sampling points (circles).

Florida (Fig. 1). It has been reported that about 4.1 to 5.6 m<sup>3</sup>/s of domestic and industrial wastewater, mainly from the City of Jacksonville, is being discharged into the Cedar and Ortega rivers and finally enters the St. Johns River (Watkins, 1992; Campbell et al., 1993; Ouyang et al., 2002). Approximately one-third of the land use in the basin is residential, while the rest of the basin consists of commercial and/or industrial, agricultural, wetland, and forest land uses. Several industrial complexes comprised of petrochemical, electrical, and plastic industries are located in this basin. Both water quantity and quality problems were observed in the Cedar and Ortega rivers basin (Bergman, 1992; Alexander et al., 1993; Ouyang et al., 2002).

#### Sampling and Sample Analysis

Grab and core sediments were collected from 58 locations along the rivers during the period between February 1998 and February 1999 by the St. Johns River Water Management District staff. A global positioning system was employed to identify each location precisely following a modified version of the USEPA's Estuarine Monitoring and Assessment Program random sampling protocols (Summers et al., 1991). The materials (such as stainless steel dredges and spoons, cellulose butyrate tubes, and glass sample collection containers) and the procedures used to collect the sediment samples have been demonstrated to be appropriate for collecting trace-level contaminant samples (Peven and Uhler, 1993a, 1993b).

**Table 1. Major kriging parameters used in this study.**

| Parameter                                    | $\gamma$ -Chlordane | $\alpha$ -Chlordane | <i>trans</i> -Nonachlor |
|--|---------------------|---------------------|-------------------------|
| Number of lags                               | 10                  | 12                  | 12                      |
| Number of data                               | 101                 | 101                 | 101                     |
| Model type                                   | spherical           | spherical           | spherical               |
| Nugget effect, $\mu\text{g/kg}$              | 0.84                | 0.91                | 0.6                     |
| Range, m                                     | 3309                | 4285                | 4285                    |
| Sill, $\mu\text{g/kg}$                       | 0.53                | 0.65                | 1.05                    |
| Mean square error, $\mu\text{g/kg}$          | 0.013               | 0.58                | 0.56                    |
| Standard mean square error, $\mu\text{g/kg}$ | 0.48                | −0.01               | −0.04                   |

Surface sediment grab samples were obtained using pre-cleaned stainless steel petite Ponar dredges. Precleaned glass trays were used to catch and retain surface dredge samples for processing and transfer. Stainless steel spoons were used to scoop the sediment samples from the glass trays into pre-cleaned 500-mL glass jars with Teflon lids. Subsurface sediment core samples were collected using piston-core samplers, following the design developed by Livingstone (1955) and modified by Fisher et al. (1992). Piston-core samplers were used to obtain undisturbed sediment samples. After collection, the samples were stored at 4°C and shipped on ice to Battelle Marine Sciences Laboratory (BMSL), Sequim, WA, for chlordane analyses using Method 8081M and for total organic carbon (TOC) analyses using Method 9060 (USEPA, 1986). The TOC was converted by high temperature combustion to carbon dioxide and then measured by either infrared absorbance or by conversion to methane and subsequent flame ionization. Although detailed descriptions of sample analytical procedures are beyond the scope of this study, they can be found in Durell et al. (2001).

### Kriging Analysis

Spatial distribution of  $\gamma$ -chlordane,  $\alpha$ -chlordane, and *trans*-nonachlor in the sediments from the Cedar and Ortega rivers was determined by ordinary kriging estimation using the ArcGIS geostatistical analyst tool (ESRI, 2001). The ordinary kriging is a weighted-linear-average estimator where the weights are chosen to minimize the estimated (kriged) variance. It uses data from a single data type to predict values of that same data type at unsampled locations. The details for mastering the art of kriging are published elsewhere (Cooper and Istok, 1988; American Society of Chemical Engineers, 1989; Isaaks and Srivastava, 1989; Rouhani et al., 1996; Goo-varts, 1999; Triantafyllis et al., 2001; Ouyang et al., 2003).

Kriging procedures used in this study include: (i) preliminary data analysis, (ii) data structural analysis, and (iii) kriging estimation. Before kriging estimation, descriptive statistics were performed to examine the chlordane data collected from the Cedar and Ortega rivers. Histogram plots of the data showed that the three chlordane species were abnormally distributed, and therefore a natural logarithmic transformation of the data was performed. In general, a normal distribution requirement in kriging analysis may not be so critical, but when the data set is too skewed or contains outliers, some kind of transformation is needed.

A data structural analysis was performed to determine the spatial correlation of the chlordane data, including experimental variogram, structural variogram model, and cross-validation analyses. The experimental variogram is an inverse measure of the two-point covariance function for a stationary stochastic process. A variogram map was constructed to determine if the spatial correlation structure of the chlordane data is dependent on direction. Because the spatial correlation distribution of the chlordane data did not apparently depend on direction, an isotropic spherical model was selected to fit the experimental variogram. The model fitting procedure was per-

formed graphically to find a structure that would be as close as possible to the experimental variogram curve.

Cross-validation is a general procedure that checks the compatibility between a set of data and a structural model. The difference between the measured value and the cross-validation estimated value is the estimation error, which gives an indication of how well the data value fits into the neighborhood of the surrounding data values. The cross-validation standardized errors between −2.5 and +2.5 represent robust data and indicate that a model can correctly predict the estimated values. In this study the cross-validation standardized errors are −0.01, 0.48, and −0.04  $\mu\text{g/kg}$ , respectively, for  $\alpha$ -chlordane,  $\gamma$ -chlordane, and *trans*-nonachlor, indicating adequacy of the models and of the parameters.

Spatial distribution of the three chlordane species was determined by the GIS geostatistical analyst tool. The kriging domain used in this study was  $3000 \times 6000$  m, which encompassed the entire Cedar and Ortega rivers. The kriging grid intervals were  $\Delta X = 100$  m and  $\Delta Y = 100$  m. Table 1 presents the major kriging parameters used in this study. It should be noted that the sediment samples were collected from the rivers, while the kriging domain has been extended from the river to the nearby land surface. This was done because the GIS geostatistical analyst tool requires a rectangle kriging domain. This raised the question regarding the acceptability of using kriging to estimate chlordane contents in areas outside the riverbed, because no soil samples were collected from the land surface. In this study, the kriging neighborhood search radius used was about 200 m, which was much less than the modeled variogram ranges (Table 1). The range is the maximum distance over which the data exhibit spatial correlation. The cross-validation standardized errors of −0.01 for  $\alpha$ -chlordane, 0.48  $\gamma$ -chlordane, and −0.04  $\mu\text{g/kg}$ , and *trans*-nonachlor further confirmed that the kriging estimations were acceptable although the kriged domain has been extended to the nearby land surface. In addition, the kriging-estimated chlordane contents in the land surface were not used for interpretation in this study.

## RESULTS AND DISCUSSION

### Spatial Distribution of Chlordane

Spatial distributions of kriged  $\gamma$ -chlordane,  $\alpha$ -chlordane, and *trans*-nonachlor concentrations in the sediments from the Cedar and Ortega rivers are shown in Fig. 2. In general, higher chlordane concentrations were found in the Cedar River sediments as compared with those in the Ortega River. The maximum concentrations of  $\gamma$ -chlordane,  $\alpha$ -chlordane, and *trans*-nonachlor in the Cedar River sediments were 20.1, 26.3, and 19.2  $\mu\text{g/kg}$ , respectively. This occurred because the water flow velocity in the Cedar River is lower than that of the Ortega River due to the narrowness of the Cedar River. A lower water flow velocity is a favorable condition for



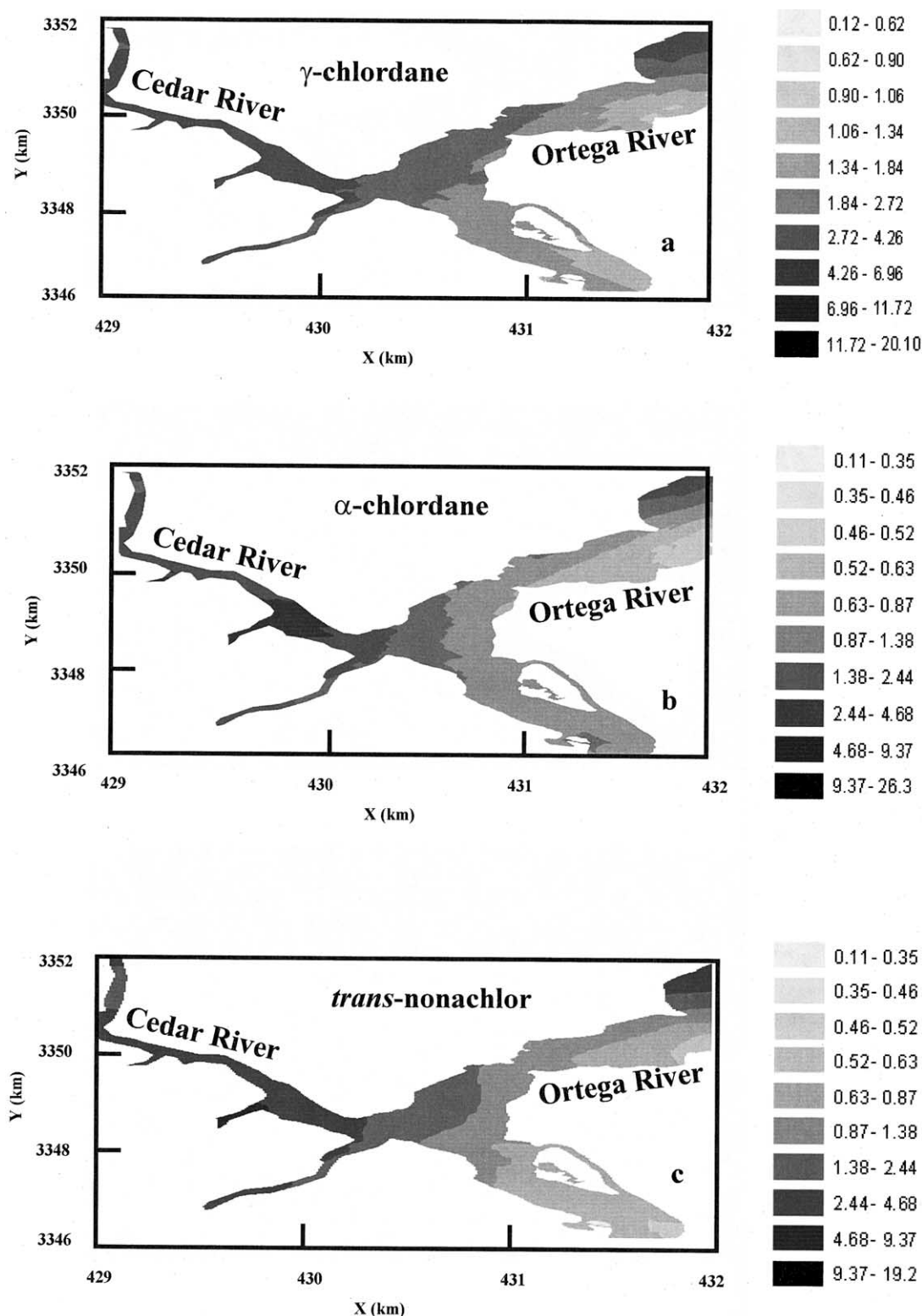


Fig. 2. Spatial distribution of kriging-estimated chlordane concentrations ( $\mu\text{g/kg}$ ).

sediment-associated chlordane accumulations. Additionally, the land uses in the area is also a factor. While the land uses in the Cedar River and in the northern end of the Ortega River are mostly residential and commercial lands, there is about 6.1% more hardwood forest and hardwood forested wetland in the southern part of the

Ortega River. Although the specific sources of chlordane contamination are unknown historically, residential and commercial lands normally contribute more chlordane sources than forested wetlands.

Figure 2 further revealed that higher chlordane concentrations also occurred at the northern end of the

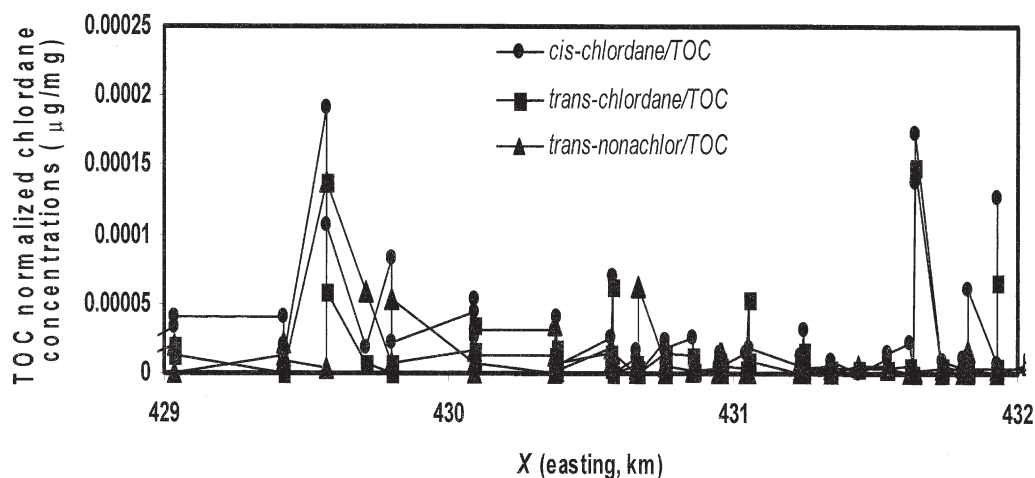


Fig. 3. Total organic carbon (TOC)-normalized chlordane concentrations as a function of distance in the  $X$  direction (easting).

Ortega River. The highest contents of  $\gamma$ -chlordane,  $\alpha$ -chlordane, and *trans*-nonachlor at this hot spot were about 15.0, 17.0, and 14.0  $\mu\text{g/kg}$ , respectively. This could be the result of an additional sediment chlordane loading from Fishweir Creek (Fig. 1) into the northern end of the Ortega River. The Fishweir Creek area has been recently identified by the St. Johns River Water Management District as a contaminated area, including contamination from chlordane and other pesticides (Durell et al., 2001).

To exclude variations due to physical properties of particles such as grain size differences and to better compare site-to-site variations in sediment chlordane contamination, normalization of sediment chlordane concentrations was performed in this study. In general, conservative elements such as Al, Fe, Se, Rb, and Li are used to normalize heavy metals, whereas organic carbon content is used to normalize hydrocarbons (Grant and Middleton, 1988).

In this study, the chlordane data were normalized with TOC. This was accomplished by dividing the chlordane concentrations by TOC concentrations at each sampling location (Fig. 3). The TOC contents were obtained from the same sediment samples as those of chlordane using Method 9060 (USEPA, 1986). Detailed description of sample analysis procedures for TOC can be found in Durell et al. (2001).

Two peaks were observed along the  $X$  direction (easting), one at  $X = 429.6$  km and the other at  $X = 431.8$  km. This finding was consistent with the kriging estimations as shown in Fig. 2. That is, the concentrations of the three chlordane compounds were high at  $X = 429.6$  km in the Cedar River and at  $X = 431.8$  km in the northern end of the Ortega River. Results indicated that the effects of grain size on sediment chlordane contamination were negligible because the normalized chlordane concentrations were consistent with those of kriging estimations. A similar result was also obtained for TOC-normalized DDT [1,1,1-trichloro-2,2-bis(*p*-chlorophenyl)ethane] at the same location (Ouyang et al., 2003), where the influence of sediment grain size or texture on DDT concentration was negligible in this river system.

### Relationships among Chlordane Species

Plots of  $\alpha$ -chlordane,  $\gamma$ -chlordane, and *trans*-nonachlor against total chlordane using linear regression and principal axis analyses are given in Fig. 4. The total chlordane consists of all of the individual chlordane compounds measured from the sediment samples. A first glance at the linear regression analysis shows that there is a very good linear correlation between  $\alpha$ -chlordane and total chlordane ( $R^2 = 0.94$ ; Fig. 4a), a somewhat linear correlation between  $\gamma$ -chlordane and total chlordane ( $R^2 = 0.71$ ; Fig. 4b), and no linear correlation between *trans*-nonachlor and total chlordane ( $R^2 = 0.02$ ; Fig. 4c). However, a residual analysis shows that the linear regression lines shown on Fig. 4 have more or less nonconstant variances and thereby could violate the following linear regression assumption: the error terms should have the same variance at each value of the predictor variable (SAS Institute, 2000). In the past, scientists often used regression lines to characterize scattergrams. These regression lines may not be appropriate for correlation analysis when both variables are random (Sokal and Rohlf, 1995).

In this study, an alternative analysis or principal axis analysis (PAA) was employed to further examine the relationships among the chlordane species. The PAA measures the nature of the relationship between two independent variables and can provide a single axis (or principal axis) to adequately represent the trend expressed by the scattergram (Sokal and Rohlf, 1995; Daughney et al., 2002). Figure 4 shows the principal axes for the chlordane species obtained using the PAA. A statistical analysis demonstrates that the slopes and intercepts of the principal axes were within the 95% confidence limits between  $\alpha$ -chlordane and total chlordane as well as between  $\gamma$ -chlordane and total chlordane, but were beyond the limits between *trans*-nonachlor and total chlordane. Results indicate that the principal axes can be used to represent the relationships (or scattergrams) between  $\alpha$ -chlordane and total chlordane as well as between  $\gamma$ -chlordane and total chlordane but not between *trans*-nonachlor and total chlordane.

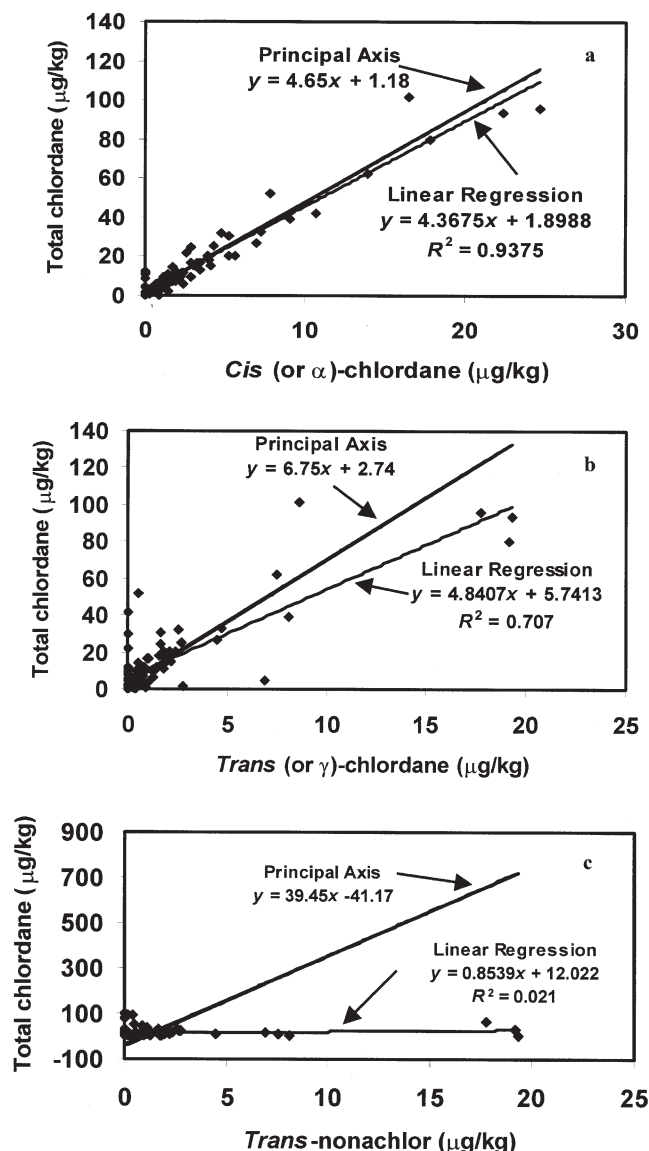


Fig. 4. Relationships between three chlordane compounds and total chlordane obtained using linear regression and principal axis analysis.

These findings are similar to the trends obtained from the regression analysis.

The correlations among the chlordane species were investigated using a *t* test with a 1% level of significance (i.e.,  $\alpha = 0.01$ ). With the *t* values of 38.32, 15.38, and 1.45, respectively, for  $\alpha$ -chlordane vs. total chlordane,

$\gamma$ -chlordane vs. total chlordane, and *trans*-nonachlor vs. total chlordane as compared with the *t* critical region (2.39), we rejected the hypothesis of zero correlation for  $\alpha$ -chlordane vs. total chlordane and  $\gamma$ -chlordane vs. total chlordane, but accepted the hypothesis of zero correlation for *trans*-nonachlor vs. total chlordane. Results imply that the degradations of  $\alpha$ -chlordane and  $\gamma$ -chlordane were in proportion to that of total chlordane in this river system although this was not true for *trans*-nonachlor. It is apparent that the metabolized pathways of *trans*-nonachlor were different from those of  $\gamma$ -chlordane and  $\alpha$ -chlordane in this river system even though the exact reasons remain unknown.

Further investigations on testing the significance of the differences between correlation coefficients among the three chlordane species were performed by a *Z* transformation of the correlation coefficients. The advantage of this transformation is that whereas correlation coefficients are distributed in skewed fashion for values of  $\rho \neq 0$  ( $\rho$  is the parametric correlation coefficient from a population), the values of *Z* are approximately normally distributed and are a basic statistical assumption for *t* test. Detailed explanations of this kind of analysis can be found in Sokal and Rohlf (1995). With the *t* values of 5.86 and 13.38, respectively, for  $\alpha$ -chlordane vs.  $\gamma$ -chlordane and  $\alpha$ -chlordane vs. *trans*-nonachlor greater than the *t* critical region (2.39), we rejected the hypothesis of equal correlation coefficients. Therefore, the differences in correlation coefficients for  $\alpha$ -chlordane vs.  $\gamma$ -chlordane and  $\alpha$ -chlordane vs. *trans*-nonachlor were significant.

### Potential Risk of Chlordane

Although kriging estimates provide useful information on spatial distribution of chlordane concentrations from sediments in the Cedar and Ortega rivers, these estimates alone do not provide an adequate basis for assessing the sediment quality problems. In this study, Florida Sediment Quality Assessment Guidelines were employed to assess the sediment quality based on the kriged chlordane concentrations.

Table 2 lists the number of samples, the mean, median, minimum, and maximum concentrations, and the standard deviations for total chlordane,  $\alpha$ -chlordane,  $\gamma$ -chlordane, and *trans*-nonachlor. The total chlordane concentrations were compared with the sediment quality assessment guideline (i.e., the threshold effect levels or TEL) for Florida coastal waters (MacDonald et al.,

Table 2. Statistical data of chlordane species in sediments from the Cedar and Ortega rivers.

| Parameter                       | Total chlordane  | $\alpha$ -Chlordane | $\gamma$ -Chlordane | <i>trans</i> -Nonachlor |
|---------------------------------|------------------|---------------------|---------------------|-------------------------|
| Number of samples               | 101              | 101                 | 101                 | 101                     |
|                                 | $\mu\text{g/kg}$ |                     |                     |                         |
| Mean                            | 13.19            | 2.58                | 1.54                | 1.37                    |
| Median                          | 6.48             | 1.01                | 0.49                | 0.45                    |
| Minimum                         | 0.02             | 0                   | 0                   | 0                       |
| Maximum                         | 101.8            | 26.3                | 20.1                | 19.2                    |
| Standard deviation              | 19.94            | 4.42                | 3.46                | 3.38                    |
| TEL†                            | 2.26             | —                   | —                   | —                       |
| Number of samples exceeding TEL | 91               | —                   | —                   | —                       |

† Threshold effect level.

1996). The value of TEL for total chlordane and the number of samples exceeding TEL are also presented in Table 2. The TEL, as defined by MacDonald et al. (1996), is the upper limit of the range of sediment contaminant concentrations dominated by no effect data entries. Within this range, concentrations of sediment-associated contaminants are not considered to represent significant hazards to aquatic organisms. The TEL can be calculated by:

$$\text{TEL} = \sqrt{\text{EDS} - L \times \text{NEDS} - M} \quad [1]$$

where EDS - L is the 15th percentile concentration in the effect data set, and NEDS - M is the 50th percentile concentration in the no effect data set. An elaborate description of the numerical sediment quality assessment guidelines and the TEL development can be found in MacDonald et al. (1996). As shown in Table 2, about 90% of the sediment samples for total chlordane had concentrations exceeding the TEL value.

Another Florida sediment quality assessment guideline is the probable effect level (PEL). The PEL, as defined by MacDonald et al. (1996), is the lower limit of the range of contaminant concentrations that are usually or always associated with adverse biological effects. Total chlordane concentrations equal to or above the PEL are considered to be hazardous to aquatic organisms. The PEL can be calculated by:

$$\text{PEL} = \sqrt{E \times NE} \quad [2]$$

where E is the 50th percentile concentration in the effect data set and NE is the 85th percentile concentration in the no effect data set. A detailed description of the numerical sediment quality assessment guidelines and the PEL development can be found in Macdonald et al. (1996).

Figure 5 shows the total chlordane distribution in the

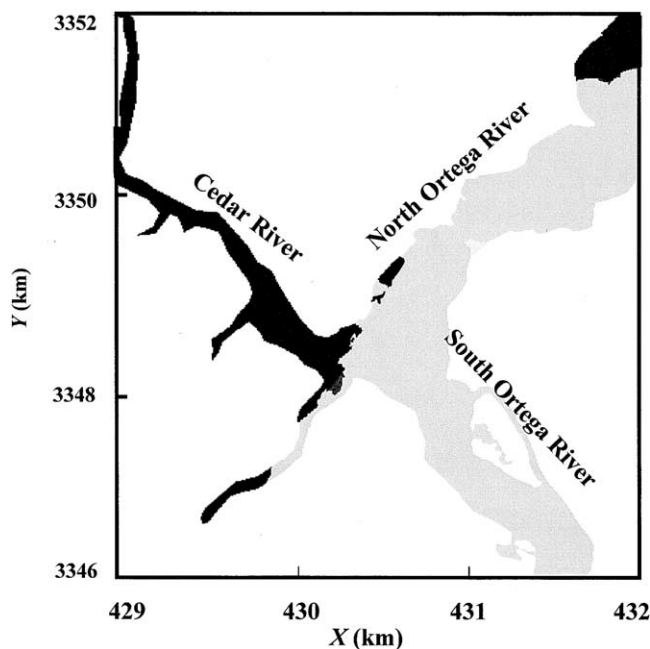


Fig. 5. Total chlordane distribution in sediments from the Cedar and Ortega rivers. Chlordane concentrations above the probable effect level (4.79 µg/kg) are displayed in black.

sediments from the Cedar and Ortega rivers with total chlordane concentrations above the PEL (4.79 µg/kg) value in dark black. It is clear from the figure that the Cedar River and the northern end of the Ortega River were found to have total chlordane concentrations above the PEL value. This could pose a potential risk to aquatic life although the concentrations do not represent the highest chlordane-contaminated spots in the environment. In our study, the highest total chlordane concentration in the river sediment is about 101 µg/kg, whereas the total chlordane concentration found in the soils from house foundations in Louisiana was up to 14 464 µg/kg (Delaplane and La Fage, 1990).

## CONCLUSIONS

Two-dimensional kriging analysis was performed to characterize spatial distribution of chlordane species in the Cedar and Ortega rivers located in the lower St. Johns River using GIS-based kriging and field measurements. In general, higher chlordane concentrations were found in the Cedar River sediments as compared with those in the Ortega River. The maximum concentrations of γ-chlordane, α-chlordane, and *trans*-nonachlor in the Cedar River sediments were 20.1, 26.3, and 19.2 µg/kg, respectively. This occurred because the average water flow velocity in the Cedar River is lower than that of the Ortega River due to the narrower span of the Cedar River. A lower water flow velocity is a favorable condition for sediment-associated chlordane accumulations.

To exclude variations due to physical properties of particles such as grain size differences and to better compare site-to-site variations in sediment chlordane contamination, normalization of sediment chlordane concentrations with TOC was performed in this study. Result indicated that effects of grain size on sediment chlordane contamination were negligible because the normalized chlordane concentrations were consistent with those of kriging estimations.

Plots of α-chlordane, γ-chlordane, and *trans*-nonachlor against total chlordane using linear regression and principal axis analyses show that good linear correlations existed among γ-chlordane, α-chlordane, and total chlordane, whereas no linear correlation existed between *trans*-nonachlor and total chlordane. Results reveal that the degradations of α-chlordane and γ-chlordane were in proportion to that of total chlordane in this river system although this was not true for *trans*-nonachlor. It is apparent that the metabolized pathways of *trans*-nonachlor were different from those of γ-chlordane and α-chlordane in this river system even though the exact reasons remain unknown. Our study further revealed that the Cedar River and the northern end of the Ortega River had total chlordane concentrations above the PEL value (4.79 µg/kg), which could pose a potential risk to aquatic life.

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